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CARBURIZED HIGH TEMPERATURE STEELS

April 1982

D. E. DIESBURG Climax Molybdenum Company of Michigan, AMAX Ann Arbor, Michigan 48106

FINAL REPORT

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ARMY MATERIALS AND MECHANICS RESEARCH CENTER Watertown, Massachusetts 02172

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ABSTRACT

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CARBURIZED HIGH TEMPERATURE STEELS Final Report - December 28, 1981 D. E. Diesburg

ABSTRACT

A detailed fracture toughness evaluation before and after a 1000-hour treatment at 315 C (600 F) of candidate steels for use at elevated temperatures in the carburized condition showed that CBS1000 offered the best combination of toughness and retention of hardness at elevated temperature. The toughness of X2(M) and X-53 decreased by about 50% during the 1000-hour treatment while that of CBS1000 changed very little. A separate study comparing six experimental steels concluded that a composition similar to that of CBS1000 had the highest impact fracture strength, even higher than that of SAE 9310.

INTRODUCTION

The National Research Council, Materials Advisory Board, has prepared Report NMAB-351 describing the shortcomings of several candidate steels for use as carburized gears for high temperature service particularly aimed for helicopter power transfer systems. One concern is the fracture resistance of steels exhibiting good hardness at elevated temperature. A preliminary investigation conducted by Climax Molybdenum using a simple impact test of a gear tooth specimen indicated that X2(M), X-53 and CBS1000, steels designed to maintain hardness at 315 C (600 F), all exhibited no loss in fracture resistance resulting from 1000-hour exposure at this temperature. Unfortunately, the initial case hardness of these steels was only HRC 51 to 55, lower than the desired minimum of HRC 58. A subsequent modification in heat treatment for all three steels was necessary to produce the desired hardness, but the fracture resistance of the steels with this case hardness has not been determined.

The goal of the present investigation was to determine metallurgically (1) where the steels mentioned, along with other helicopter gear steels used for lower temperature service, obtain their fracture resistance, and (2) how the level of fracture resistance changes when the alloys are exposed to service temperatures. The program determined the residual stress distribution and fracture toughness gradients in the carburized cases of SAE 9310, X2(M), X-53, CBS1000, and CBS600 before and after 1000-hour exposure to 315 C (600 F). After analyzing these results, alloy modifications designed to overcome any shortcomings of existing steels were prepared and tested. The fracture behavior of the experimental steels was compared to that of SAE 9310.

EXPERIMENTAL PROCEDURES

Candidate Commercial Steels

All the alloy steels evaluated in the initial portion of this program were obtained commercially either from steel producers or from gear manufacturers. Each steel was specified to be of highest quality, similar in quality to steels specified for helicopter gearing. All the steels were chemically analyzed to verify the analyses supplied with the steels.

The steels were supplied as bar stock of various diameters. The stock was heated to 1200 C (2200 F) and forged to 15 mm (0.6 in.) square bars. Thirty specimens for fracture toughness testing, 10 by 10 by 50 mm (0.4 by 0.4 by 2 in.), were machined from each steel along with carbon gradient bars. The carbon gradient bars were carburized along with test specimens in a Leeds and Northrup microcarb controlled atmosphere pit furnace. The carbon profiles present in the carburized cases were determined from carbon gradient bars. Carburized bars were softened by tempering at 540 C (1000 F) for at least one hour, and chips were machined in incremental layers for carbon analysis by a

combustion method. The first five layers were removed in $0.13~\mathrm{mm}$ $(0.005~\mathrm{in.})$ increments while the latter five were removed in $0.25~\mathrm{mm}$ $(0.010~\mathrm{in.})$ increments.

After carburizing, the unnotched fracture toughness specimens were hardened and tempered using recommended heat treat procedures outlined in Appendix A. After hardening, half of the specimens were given a 1000-hour exposure to 315 C (600 F).

Hardened and heat treated specimens were notched to various depths using an electrodischarge machine (EDM). The location of the notches was the same as that used for normal Charpy V-notch specimens (see ASTM E23 Standard Testing Procedure). The EDM notches ranging in depth from 0.05 to 1.0 mm (0.002 to 0.040 in.) were sharpened by fatigue precracking. The fatigue precracking was accomplished by cycling between a constant maximum and minimum load, where $P_{\text{max}}=10\ P_{\text{min}}$. The best control of the precracking procedure was obtained with $P_{\text{max}}=360\ \text{kg}$ (800 lb), although it was necessary to increase P_{max} to 725 kg (1600 lb) for the specimens having the shortest EDM notches. The number of cycles required for successful precracking ranged from 30,000 cycles for the specimens with the long EDM notches to 250,000 cycles for the specimens with the short EDM notches.

Once precracked, the specimens were broken in three-point bending as specified in ASTM E399. The load and displacement across the notch opening for each test were recorded. The fracture toughness was calculated using the equation for bend specimens in ASTM E399.

Representative specimens from each steel in both heat treated conditions were used for metallographic and x-ray diffraction examinations. The microstructures were examined both optically and with an AMR 1000 scanning electron microscope (SEM) equipped with an energy dispersive analyzer for x-rays (EDAX). The EDAX equipment was used to qualitatively compare the composition of carbides and/or other precipitates before and after the 1000-hour exposure to 315 C (600 F).

Profiles of residual stress and retained austenite content in the cases of the commercial candidate steels were determined by x-ray diffraction techniques described in detail elsewhere. The surfaces of the bend specimens were sequentially electropolished with perchloric ethanol (78 cc perchloric acid, 100 cc butylcellosolve, 120 cc water and 700 cc ethanol) at a 40 V applied potential. A Rigaku x-ray diffraction system equipped with a CrK_α x-ray source was used. The multiple-exposure method, or the $\sin^2\psi$ technique, was employed -- four exposures for martensite stress determination and five for austenite in each of the positive and the negative ranges of ψ -angles. Therefore, the total number of exposures was nine for the martensite and eleven for austenite. The ψ -angles were chosen so that $\sin^2\!\psi$ values would vary by 0.1 for the austenite 220 peak from 0 up to 0.5, and by 0.15 for martensite 211 peak to 0.6. The retained austenite content was determined from the ratio of austenite 220 and martensite 311 peak intensities. The intensity of each peak was the average value of intensities at various ψ -angles which were corrected for absorption due to ψ -tilt.

The hardness of the carburized cases was evaluated both with HRA and microhardness (HV 0.5) techniques. The HRA was determined directly on the surface and converted to the more common HRC hardness values. Microhardness traverses were made across the case on a polished surface perpendicular to the carburized specimen surface. Impressions were made with a 500 g load in increments of 0.13 mm (0.005 in.) to a depth of 0.76 mm (0.030 in.) below the surface and continuing in increments of 0.25 mm (0.010 in.) to a depth of 2.29 mm (0.090 in.). From these hardness profiles, the distances from the specimen surface to the location in the case where the hardness is 510 DPH were estimated. This hardness value is equivalent to HRC 50, and the distance to 510 DPH is referred to as the "effective case depth."

Experimental Steels

After evaluating the results from the commercially produced steels, six experimental steels were prepared. Both fracture strength (not fracture toughness) and the hardness after tempering at 315 C (600 F) of all six experimental steels were evaluated and compared to that of SAE 9310. The experimental steels were prepared as three 30 kg (66 lb) induction melted heats, each heat split to produce two compositions. The heats were melted under an inert atmosphere of argon and poured into 89 mm (3-1/2 in.) diameter ingots 203 mm (8 in.) in length. A chemical analysis was obtained for each steel. The SAE 9310 steel used in this part of the program was commercially produced as 51 mm (2 in.) bar.

The 89 mm (3-1/2 in.) diameter ingots and the SAE 9310 steel were heated to 1200 C (2200 F) and forged to 32 mm (1-1/4 in.) diameter bar. Both impact fracture strength specimens and carbon gradient bars were machined from the forged bar. The dimensions of the impact fracture strength specimen are shown in Figure 1. The carbon gradient bars were carburized along with the machined test specimens and used as described previously for determining the carbon profile of the carburized cases. The carburizing and heat treatment cycles given to these steels are given in Appendix B. After carburizing, half of the experimental steel specimens were heated to 315 C (600 F) for 1000 hours.

Impact fracture strengths of carburized specimens before and after the 1000-hour temper at 315 C (600 F) were determined using a Riehle impact machine equipped with an instrumented Izod striker. Each end of the specimen (Figure 1) was tested individually by securing the specimen in the anvil of the impact machine in a manner similar to securing Izod test specimens as described in ASTM E23. The instrumented striker measures the maximum load required for fracture which is used to calculate a bending stress. This stress is defined as the impact strength of the carburized case. Such a test is particularly useful because it evaluates the impact behavior of the case without measuring total energy absorbed which can be dominated by a high toughness in the core.

The ability of the experimental steels to resist a number of repeated low energy impacts was also evaluated. Past research³ has shown that the ability of carburized steels to resist repeated low energy impacts can be

correlated to the impact strength of the carburized case, as measured using the instrumented impact test described above. Repeated impact tests are performed using the same procedure as that used for the impact fracture strength test except the drop height of the hammer is very low, a height corresponding to an energy impact of 4 J.

Surface hardness of the carburized specimens was evaluated before and after 1000 hours at 315 C (600 F) by taking HRA measurements directly on the carburized surface and converting the readings to HRC values. Hardness gradients of carburized cases were determined from representative specimens before and after the 1000 hour exposure. No change in hardness after exposure was taken as an indication of good microstructural stability.

The hot hardness of each carburized steel was determined between room temperature and 371 C (700 F). Vickers hardness impressions were made directly on the carburized surface with a 2.5 kg load on specimens heated in 55 C (100 F) increments starting at 93 C (200 F) until reaching 371 C (700 F). The surface of the specimens had been polished with 600 grit paper, removing not more than $0.05~\mathrm{mm}$ ($0.002~\mathrm{in.}$), prior to hot hardness testing.

The microstructures of the carburized cases of the experimental steels were examined optically and using a scanning electron microscope (SEM).

RESULTS

The chemical analyses of both the candidate commercial steels and the experimental steels are given in Table 1. All commercially produced steels were found to be within specifications. No difficulty was encountered in forging or machining. Carbon analyses of the carburized cases are shown in Figures 2a and b. The case hardness values taken before and after 1000 hours at 315 C (600 F) are shown in Table 2. All the candidate commercial steels exceeded the specified aim of HRC 58 before the 1000-hour exposure. Two experimental steels (C and E), both containing 2.51% Cr, were too soft in the hardened and tempered condition before exposure to 315 C (600 F). The 9310 composition had a hardness less than HRC 58 after the 1000-hour exposure to 315 C (600 F).

The microhardness traverses of the carburized cases were used to determine the effective case depth which is defined in this study as the distance to a hardness of HV 510. The effective case depths are shown in Table 3.

The hot hardness results from the experimental steels are plotted in Figure 3 as are results obtained from the commercial candidate steels determined in a previous study. The slopes of the curves are indications of hardness retention at elevated temperature. All the experimental steels exhibit similar and adequate hardness.

Figure 4 shows the microstructures of the commercial candidate steels before and after 1000 hour exposure at 315 C (600 F). All the microstructures were martensitic with varying amounts of retained austenite and dispersed carbide. The carbides present in the microstructure of SAE 9310 and CBS600 were contained in the outer 0.05 mm (0.002 in.) of the carburized surface. All the carbides were dispersed evenly through the grains of the commercial steels except that of CBS600 which exhibited grain boundary carbides in the outer 0.05 mm (0.002 in.) of the case. The microstructures of the carburized experimental steels are shown in Figure 5. Four of the six experimental steels (A, B, C, E) exhibited a network of carbides in the prior austenite grain boundaries in addition to a fine distribution of carbides within the grains. Two experimental steels (D and F) showed only a fine distribution of carbides within the grains and no carbide network in the prior austenite grain boundaries.

The microstructures of the five commercial candidate steels were examined using a scanning electron microscope. The carbides were qualitatively analyzed with an energy dispersive analyzer (EDAX). A wave length scan of the x-rays from various carbides within the microstructures of three commercial candidate steels (CBS1000, X2(M) and X-53) was obtained and the results are summarized in Table 4. These three steels were the only steels that strengthened by precipitation during tempering, and it was important to determine the change in carbide composition of these steels. The carbides of the other steels showed no noticeable change in composition resulting from the 1000-hour exposure at 315 C (600 F).

Table 4 shows that CBS1000 exhibited no change in carbide composition or size during the 1000-hour treatment at 315 C (600 F). The X2(M) steel indicated precipitation of complex carbides containing vanadium that were not present before the 1000-hour treatment. The carbides in the X-53 steel were not as complex in composition as those for CBS1000 and X2(M) and only contained Fe and Mo. Precipitates containing Cu and/or Ni were not observed in the X-53 steel before or after the 1000-hour treatment.

The amounts of retained austenite at several locations in the carburized cases of the five commercially produced steels were determined by x-ray diffraction and are plotted in Figure 6. Measurements were made at the various locations within the carburized cases by electrochemically polishing away the outer regions of the cases. Before the 1000-hour temper at 315 C (600 F), all five steels showed the presence of retained austenite ranging from less than 15% in CBS1000 to over 25% for X-53. After the 1000-hour exposure, the retained austenite in SAE 9310 and CBS600 was reduced to less than 2% while that of CBS1000, X2(M) and X-53 remained essentially unchanged. The microstructures of CBS1000, X2(M) and X-53 are very stable at 315 C (600 F), even with respect to the amount of retained austenite in the case.

The same polished surfaces used for retained austenite measurements were used for residual stress measurements. Residual stress values were corrected for the effect of the surface removal. Residual stress gradients in the carburized cases before and after the 1000-hour temper at 315 C (600 F) are shown in Figure 7. The 1000-hour temper reduced the compressive residual stress in the SAE 9310 and CBS600 steels almost to zero. The residual stress profiles of CBS1000, X2(M) and X-53 changed less dramatically than that of SAE 9310 and CBS600 because of the heat treatment, again an indication of the stable microstructure.

Figure 8 shows that the residual stress in the retained austenite in the CBS1000, X2(M) and X-53 steels is tensile. Oftentimes refrigeration treatments result in tensile residual stresses in retained austenite. All three of these steels had been given a refrigeration treatment. Such tensile residual stresses have been observed to cause poor fatigue limits in high-cycle fatigue tests.

Fracture toughness gradients for each carburized case before and after the 1000-hour temper are shown in Figure 9. These curves have been corrected for the residual stress contribution to measured values. Because of the strong influence of carbon on fracture toughness and the large variation in carbon profiles in the carburized cases shown in Figure 2, it was necessary to plot fracture toughness against carbon content as was done in Figure 10. The fracture toughness values at 0.5 and 0.75% C were determined from Figure 10 and are listed in Table 5. The candidate steels intended for elevated temperature service decreased in toughness after the 1000-hour treatment at 315 C (600 F). The SAE 9310(1) steel softened considerably by the 1000-hour treatment and, as expected, the fracture toughness tended to increase as shown in Figure 9.

The impact fracture strengths of the experimental steels a prompared to that of SAE 9310(2) in Table 6. The experimental steels were tested before and after the 1000-hour treatment at 315 C (600 F). All steels were tested in the as-carburized condition with no grinding of the carburized surface. The impact fracture strengths of Steels D and F were the only values to exceed that of SAE 9310. These two steels were the only experimental steels that did not exhibit a network of carbides in the prior austenite grain boundaries, as mentioned earlier. The grain boundary carbides are believed to have contributed to the relative low fracture strength of Steels A, B, C and E.

Table 6 also contains the number of repeated low-energy impacts required to completely fracture the specimens. Because of the great scatter in data expected in this test, the individual counts are tabulated rather than the averages. It must be pointed out that these values can be highly dependent on the properties of the core. Cracks can occur in the case early in the test and still require many impacts to propagate completely through the specimen. One specimen of SAE 9310 was impacted once, sectioned, and examined for evidence of case cracking. A crack was observed, indicating that even in a steel capable of withstanding 400 to 500 impacts before complete fracture, the cracking process begins with the first impact. Specimens of Steels D and F did not crack early in the test, even after receiving 25 impacts no cracks were observed. Some of the specimens of three of the experimental steels (A, B and C) broke completely with one low-energy impact.

DISCUSSION

This research program was performed in two parts. The first part was to determine, in terms of fracture toughness and residual stress, the behavior of four commercially produced steels intended for elevated temperature service in the carburized condition. The second was to produce experimental steels and evaluate their general fracture properties in impact.

Fracture Toughness of Commercial Candidate Steels

The aim fracture toughness in the carburized cases is given in Figure 10 by the gradient for SAE 9310 before the 1000-hour treatment at 315 C (600 F). The behavior of SAE 9310 after the 1000-hour treatment is only of academic interest because the case hardness of this steel becomes too low at this temperature, which is why SAE 9310 cannot be used at elevated temperatures. It would be ideal if one of the candidate elevated temperature steels would exhibit the same room temperature toughness as SAE 9310 and maintain this level of toughness even after 1000 hours at temperature. Table 3 shows that none of the candidate steels tempered 1000 hours at 315 C (600 F) matched the room temperature toughness of SAE 9310. The fracture toughness values before the 1000-hour temper shown in Figure 10a indicate that the case fracture toughness of all the candidate steels is about equal to that of SAE 9310. However, the toughness of CBS1000, X2(M) and X-53 decreased after the 1000-hour temper, with that of CBS1000 decreasing the least amount (Table 5). The toughness of CBS600 decreased slightly but remained above the aim toughness of SAE 9310.

The fracture toughness values shown in Figure 10 have the residual stress contribution removed from consideration. Figure 11 is a similar plot but contains the contribution of residual stress, including the tensile stress in the austenite. Since retained austenite represented a sizable portion of the case microstructure of the X-53 steel, the tensile nature of residual stress in this phase had a significant effect on the measured fracture toughness, as incicated in Figure 11 by the relatively low fracture toughness for this steel. Figure 10 is a comparison of the fracture toughness potential of the steels because the curves have been corrected for differences in residual stress. Figure 10 shows that X-53 has slightly more fracture toughness than X2(M) in the high carbon region suggesting that if the retained austenite were not present or if the residual stresses were not tensile, then X-53 would offer at least as much or slightly more fracture toughness than X2(M). Neither X-53 nor X2(M) had fracture toughness as high as CBS1000, and none of the three steels exhibited the aim fracture toughness after the 1000-hour temper.

The fracture toughness results indicate that, of the three candidate steels that exhibit good hardness retention and microstructural stability at 315 C (600 F), the best is CBS1000 followed by X-53 and then X2(M). The X-53 steel is judged better than X2(M) only if the retained austenite can be kept at a relatively low level. Both X2(M) and X-53 exhibited a large decrease in fracture toughness (Table 5), whereas CBS1000 showed a toughness decrease of

only 10% at 0.50% C, and actually increased in toughness at 0.75% C. Because of the small decrease (if any) in fracture toughness, CBS1000 appears to be the best choice for elevated temperature service. CBS1000 may have less than the aim fracture toughness, but the toughness will not decrease during service and therefore its performance should be stable and predictable.

The compressive residual stress in the carburized case of CBS1000 never exceeded 140 MPa (20 ksi), as shown in Figure 7. Such a low compressive residual stress could be caused by the small section size of the test specimen relative to the level of hardenability of this steel. A larger section may allow a greater compressive stress to be developed in the case.

Experimental Steels

Based on the fracture toughness and residual stress evaluation of the commercial candidate steels, it was concluded that the precipitation strengthening characteristics of X-53 and X2(M) were too strong and that the fracture toughness of the microstructure decreased after exposure to elevated temperature. All the candidate steels lost most of their residual compressive stresses in the case during the exposure. This fact probably cannot be overcome by alloy modification; therefore, the only benefit to be gained by alloy modification is to obtain a steel that does not simultaneously lose fracture toughness. Rather than to evaluate compositions that develop elevated temperature strength through precipitation, it was decided to evaluate steels that have a high degree of temper resistance, steels containing high molybdenum and vanadium. Molybdenum and vanadium are known to improve elevated temperature strength as demonstrated by the performance of certain tool steels and bearing steels. A commonly used bearing steel (M-50) contains 1.3% V and 4.2% Mo. It was decided to base the alloy selection of the experimental steels around 1.2% V and 2.2% Mo. The chromium content of four of the six steels was kept at 1.0% in an attempt to improve the toughness and carburizing characteristics. Past research on carburizing steels indicates that chromium tends to decrease toughness unless combined with nickel; 5 therefore, nickel was added to two of the steels containing 1.0% Cr. There also has been an observed toughness benefit for combining nickel and molybdenum in carburizing steels. Two steels having 2.5% Cr were included because it was not certain whether the steels containing 1.0% Cr would maintain enough elevated temperature hardness. Silicon is known to improve the temper resistance of steels, especially at carbon levels corresponding to the core. The temper resistance offered by silicon becomes less as the carbon content increases. Because core strength is important in carburized gears, five of the six experimental steels contained 1.0% Si. The base composition is represented by Steel A while Steel B was intended to evaluate the effects of adding molybdenum, Steel C the effect of adding chromium, and Steel D the effect of adding nickel. Steel E was a low silicon modification of Steel C and Steel F was a low vanadium modification of Steel D.

Table 6 shows that only the alloy combination of nickel (2.0%) and molybdenum (2.3%) produced steels (Steels D and F) that had fracture strengths greater

than that of SAE 9310. The 1000-hour exposure to 315 C (600 F) actually increased the fracture strength of Steels D and F, even though there was very little change in surface hardness (Table 2). It should also be noted that the effective case depths of both Steels D and F were greater than that of SAE 9310 and that of the other experimental steels. This fact makes Steels D and F look even better, because an increase in case depth usually decreases impact fracture strength. 6

Comparing Steel D and Steel F, Steel F contained 0.6% V rather than 1.2% and it had the higher fracture strength after the 1000-hour treatment. These results favor the lower vanadium addition. The carbide distribution in the microstructure of both steels can be seen in Figure 4. No carbide networks were observed in the prior austenite grain boundaries as were observed in the other four experimental steels.

Comparing Steels C and E indicates that the lower silicon results in a slight improvement in fracture strength; however, 1.0% Si may still be necessary to maintain core hardness at elevated temperatures.

The slopes of the hot hardness curves in Figure 3 can be used to evaluate the relative elevated temperature strengths of steels. All the experimental steels had slopes similar to those of the commercial candidate steels CBS1000, X2(M) and X-53. This similarity suggests that all of the experimental steels offer as much elevated temperature strength as the commercial candidate steels.

Combining all the above observations Steel F seems to represent the experimental steel offering the best combination of fracture strength and hardness retention at elevated temperature. The composition of this steel is very close to that of the commercially available CBS1000 steel which was the steel recommended from the fracture toughness portion of this study. The major differences between Steel F and CBS1000 is that Steel F contained less nickel and molybdenum. The CBS1000 steel also contained less silicon.

SUMMARY

A fracture toughness comparison of three commercial steels CBS1000, $X2\,(M)$ and X-53 showed that CBS1000 offered the bost combination of toughness and retention of hardness at elevated temperature. The fracture toughnesss in the carburized cases of both X2(M) and X-53 decreased by about 50% when held at 315 C (600 F) for 1000 hours. That of CBS1000 changed very little. A separate comparison of six experimental steel compositions concluded that a composition very similar to that of CBS1000 exhibited the best impact fracture strength. The experimental steel contained less nickel and molybdenum than CBS1000, 2.0% vs. 3.0% and 2.3% vs. 4.2%, respectively. The optimum nickel-molybdenum combination may be somewhere between these values.

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Table 1

Analysis of Steels

						We	Weight,	%					ndd	Ħ
Steel	ပ	Μ'n	Si	Cr	Ni	Мо	3	Cu	Λ	A1	ď	S	В	z
Commercial:														
CBS600	0.19	0.62	0.98	1.48	NDa	0.99	QN	QN	Q	0.040	0.008	0.004	5	34
CBS1000	0.14	0.58	0.52	1.00	3.00	4.17	QN	ON	0.37	0.056	0.010	900.0	<u>\$2</u>	95
X2 (M)	0.16	0.36	0.94	4.72	QN	1.23	1.34	QN	0.42	0.010	0.015	0.005	~	58
x-53	0.11	0.38	1.00	1.00	1.98	3.15	Q.	1.99	QN	<0.005	0.009	0.003	<u>≤2</u>	71
SAE 9310(1) ^b	0.11	0.68	0.28	1.25	3.49	0.13	QN	QN	QN	0.028	0.012	0.005	7	62
SAE 9310(2) ^C	0.13	0.48	0.24	1.22	3.15	0.11	QN	0.12	QN	ND	0.010	0.016	QN	Q.
Fxnerimen-														
tal Heat:														
A (P2622A)	0.14	0.53	1.18	1.00	!	2.20	{	1	1.19	0.013	0.012	0.012	1	53
B (P2622B)	0.14	0.53	1.07	1.00	! }	3.28	1	!	1.19	0.003	0.012	0.012	1	50
C (P2623B)	0.14	67.0	1.05	2.51	ł	2.31	1	1	1.16	0.004	0.012	0.012	1	59
D (P2624B)	0.14	0.50	1.06	1.04	2.02	2.32	1	1	1.18	0.008	0.011	0.014	1	99
E (P2623A)	0.14	0.49	09.0	2.51	1	2.31	!		1.16	0.012	0.012	0.012	1	99
F (P2624A)	0.14	0.50	1.10	1.04	2.02	2.32	1		0.62	0.005	0.011	0.014	1	54

 $a_{\rm ND} = {\rm Not \ Determined.}$

^bSteel involved in fracture toughness and residual stress portion of program.
^cSteel compared to the experimental steel.

Table 2
Surface Hardness of Carburized and Hardened Steels

	Hardnes:	s, HRC ^a
<u>Steel</u>	Before Exposureb	After Exposureb
CBS600	65.4	58.8
CBS1000	58.6	58.4
X2 (M)	58.7	61.0
X-53	58.4	59.5
SAE 9310(1)	61.2	52.3
SAE 9310(2)	60.8	$ND^{\mathbf{C}}$
A (P2622A)	59.0	58.1
B (P2622B)	59.5	59.4
C (P2623B)	55.3	56.6
D (P2624B)	59.4	59.5
E (P2523A)	54.3	54.1
F (P2624A)	59.7	59.0

^aConverted from HRA determinations.

 $^{^{}m b}{
m Exposure}$ to 315 C (600 F) for 1000 hours.

^cND = Not Determined.

 $\begin{array}{c} \underline{\text{Table 3}} \\ \\ \underline{\text{Effective Case Depth of Carburized Steels}}^{a} \end{array}$

Ste <u>el</u>	Effective Case ^b Depth, mm (in.)
CBS600	1.52 (0.060)
CBS1000	1.22 (0.048)
X2(M)	1.52 (0.060)
x-53	1.12 (0.044)
SAE 9310(1)	1.30 (0.051)
SAE 9310(2)	1.14 (0.045)
A (P2622A)	0.89 (0.035)
B (P2622B)	1.02 (0.040)
C (P2623B)	1.07 (0.042)
D (P2624B)	1.40 (0.055)
E (P2623A)	1.14 (0.045)
F (P2624A)	1.52 (0.060)

^aSteels not given 1000-hour treatment at 315 C (600 F).

 $^{^{\}mbox{\scriptsize b}}\mbox{\scriptsize Defined}$ as the distance to a hardness of HV 510.

Table 4

Summary of EDAX Analysis of Particles in Carburized Cases

		Elements Present ^a		
<u>Steel</u>	Particle	<u>Before</u> ^b	<u>After</u> ^b	
CBS1000	Small	Fe,Mo,V	Fe,Mo,V	
	Intermediate	Fe,Mo	Fe,Mo	
X2 (M)	Small	Fe,Mo,W,Cr,V Fe,W,Mo,Cr	Fe,Mo,W,Cr,V Fe,W,Mo,Cr	
	Intermediate		Fe,Cr,W,Mo,V	
	Massive	Fe,Cr,W,Mo	Fe,Cr,W,Mo,V	
x- 53	Small Intermediate	Fe,Mo	Fe,Mo Fe,Mo	

^aElements listed in order of decreasing amount (second elements listed ranged from 10 to 30%).

 $^{^{}m b}$ Before and after 1000-hour exposure to 315 C (600 F).

Table 5

Fracture Toughness in Carburized Cases (Corrected for Residual Stress Effects)

	Carbon	Fracture Toughness, K _{Ic} ^a MPa√m (ksi√in.)		
Steel	Carbon Content,	Before Exposure ^b	After Exposure	Change, c
CBS600	0.50	53 (48)	47 (43) ^d	(10)
	0.75	45 (41)	36 (33) ^d	(8)
CBS1000	0.50	44 (40)	33 (30)	(10)
	0.75	21 (19)	26 (24)	25
X2 (M)	0.50	45 (41)	21 (19)	(53)
	0.75	25 (23)	13 (12)	(48)
X-53	0.50	48 (44)	22 (20)	(54)
	0.75	36 (33)	23 (21)	(36)
SAE 9310	0.50 0.75	42 (38) 27 (25)	Too Soft Too Soft	

 $^{{}^{}a}\!\mathrm{K}_{\text{Ic}}$ determined using specimens with short crack lengths.

bExposure to 315 C (600 F) for 1000 hours.

 $^{^{\}mathbf{c}}$ Parentheses indicate the change was a decrease (.egative).

 $^{^{}m d}$ Some softening occurred but remained above HRC 58 (see Table 2).

Table 6

Impact Properties of Carburized Cases of Experimental Steels With and Without 1000-Hour Temper Compared to SAE 9310

	Impact Fracture Strength,		Number of Repeated Impacts		
	MPa (ksi)		Complete Fracture		Crack Initiation
Steel	Before ^a	After ^a	Before ^a	After ^a	Before
SAE 9310(2)	3344 (485)	$^{ m ND}^{ m b}$	453, 516	ND	1
A (P2622A)	2758 (400)	2393 (347)	10, 30	1, 1	1
в (Р2622В)	1751 (254)	1963 (285)	1, 1	1, 1	1
C (P2623B)	2544 (369)	2838 (412)	1, 7	1, 9	1
D (P2624B)	3957 (574)	4203 (609)	164, 194	83, 88	>25
E (P2623A)	2578 (374)	3129 (454)	2, 223, 295	153, 155	2
F (P2624A)	3805 (552)	4921 (713)	314, 391	216, 320	>25

 $^{^{\}rm a}{\rm Before}$ and after 1000-hour treatment at 315 C (600 F).

b_{ND} = Not Determined.

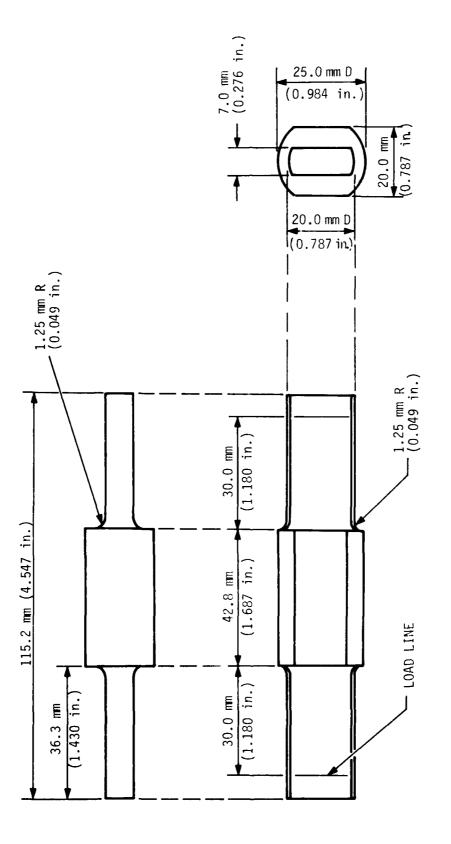


Figure 1 Specimen Geometry Used for Determining the Impact Fracture Stress of Carburized Steels

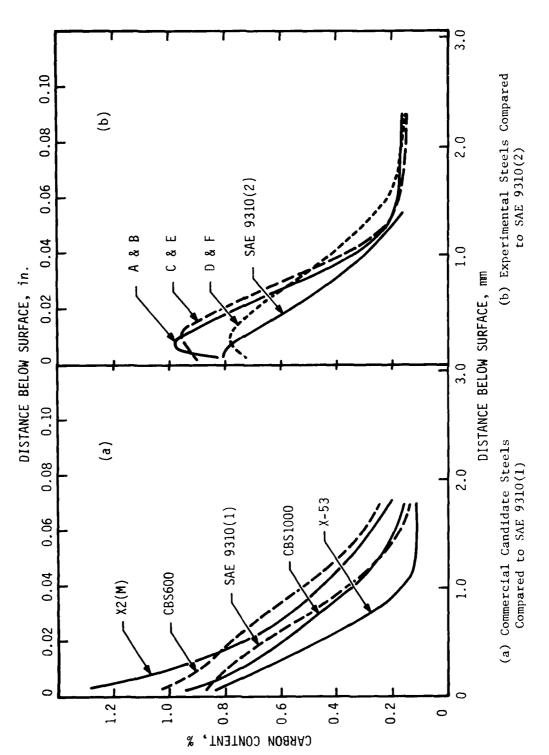


Figure 2 Carbon Profiles in Carburized Cases

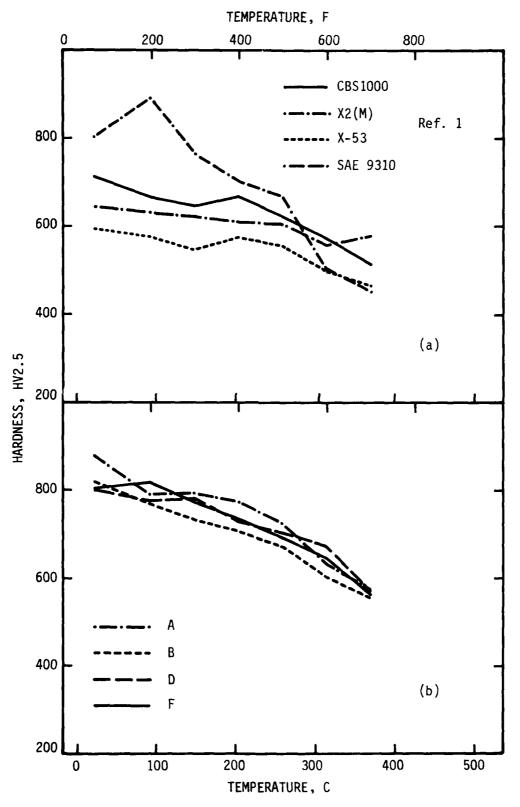
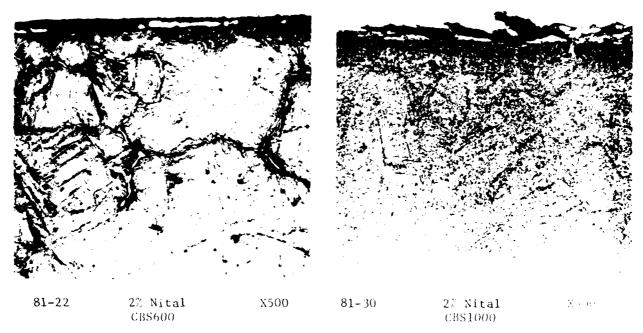


Figure 3 Hot Hardness of Carburized Steels (a) Commercial Candidate Steels from Reference 1 (CBS1000, X2(M) and X-53 did not have HRC 58 Surface Hardness) (b) Four Experimental Steels



Before 1000 hours at 315 C (600 F)

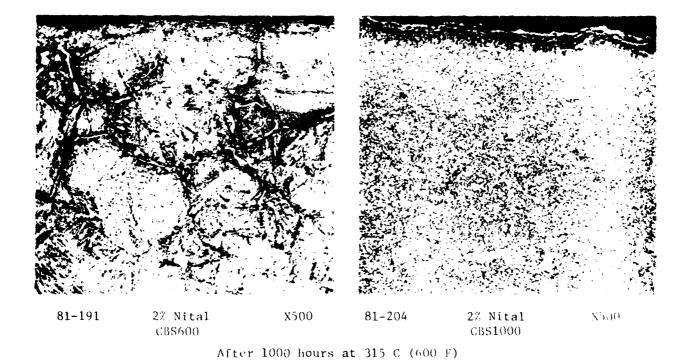
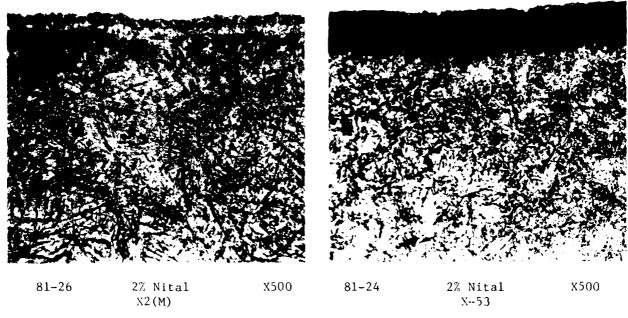
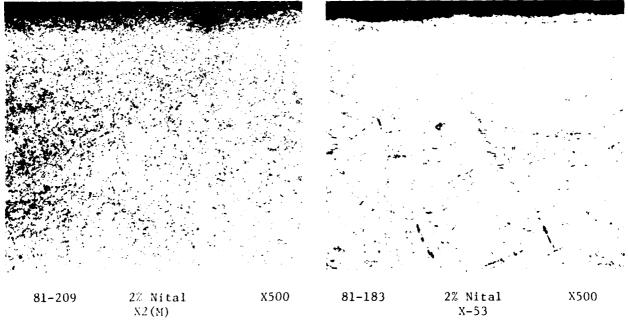


Figure 4 Microstructure of Carburized Cases Before and After $1000~\mathrm{hours}$ at $315~\mathrm{C}$ (600 F)



Before 1000 hours at 315 C (600 F)



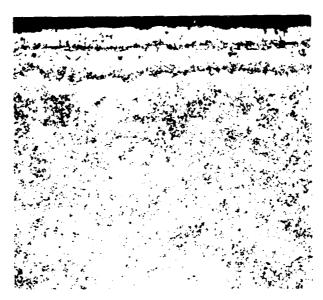
After 1000 hours at 315 C (600 F)

Figure 4 (Continued)



81-28 2% Nital X500 SAE 9310(1)

Before 1000 hours at 315 C (600 F)



81-195 2% Nital X500 SAE 9310(1)

After 1000 hours at 315 C (600 F)

Figure 4 (Continued)

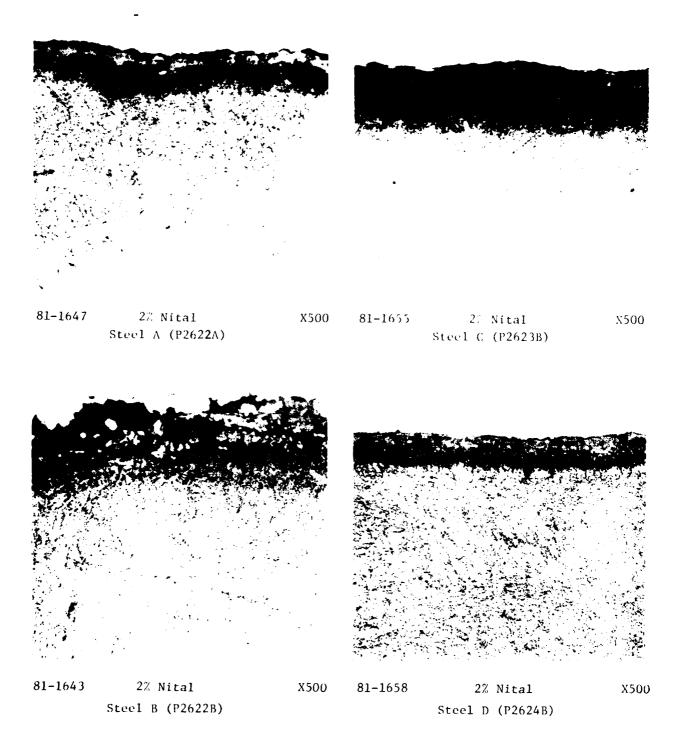


Figure 5 Microstructure of Carburized Experimental Steels (Not Tempered 1000 Hours)





81-1654 2% Nital X500 Steel F (P2624A)

Figure 5 (Continued)

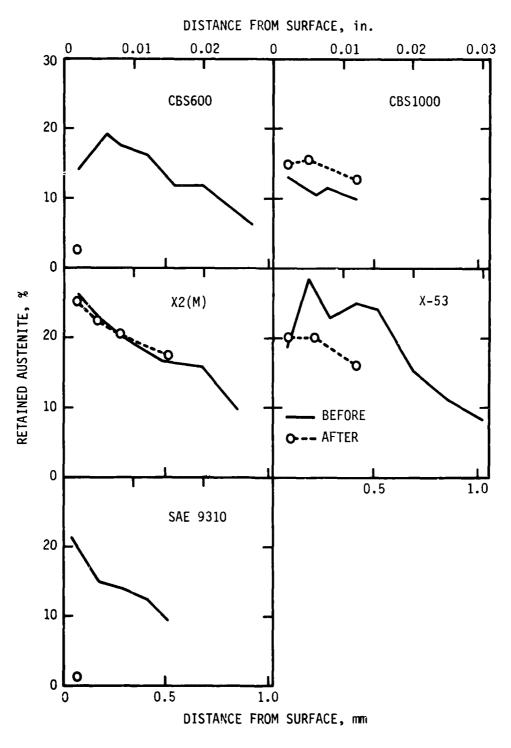


Figure 6 Retained Austenite Content of Carburized Cases
Before and After 1000 Hours at 315 C (600 F)

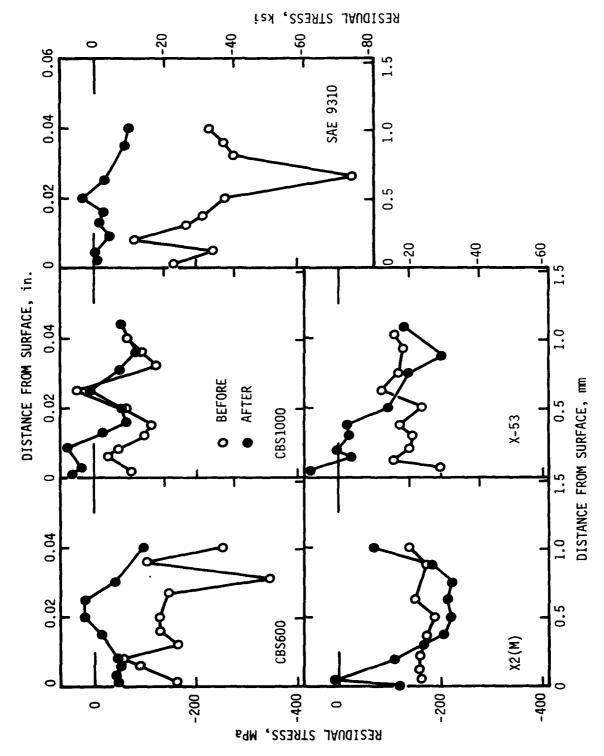


Figure 7 Residual Stress in Martensite of Carburized Cases Before and After 1000 Hours at 315 C (600 F)

Figure 8 Residual Stress in Retained Austenite after 100° Hours at 315 C (600 F)

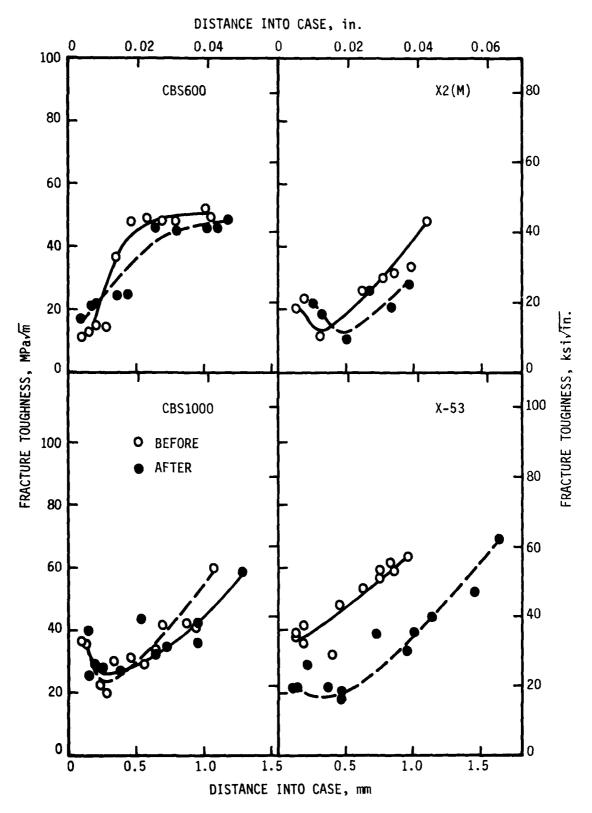


Figure 9 Fracture Toughness Gradients in Carburized Cases of Candidate Steels
Before and After 1000 Hours at 315 C (600 F)
(Corrected for residual stress effects)

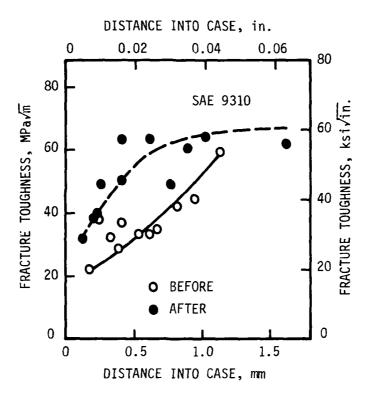


Figure 9 (Continued)

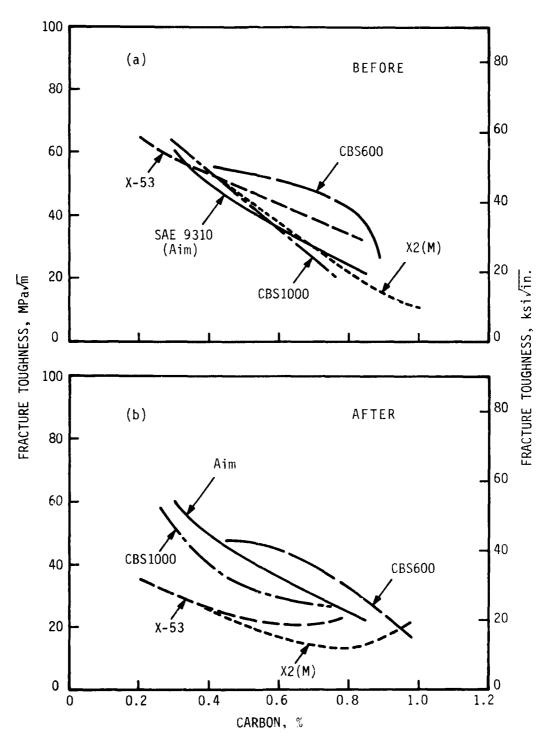


Figure 10 Fracture Toughness of Various Carbon Contents for Candidate Steels Compared to SAE 9310 (Aim). (Corrected for residual stress effects.)

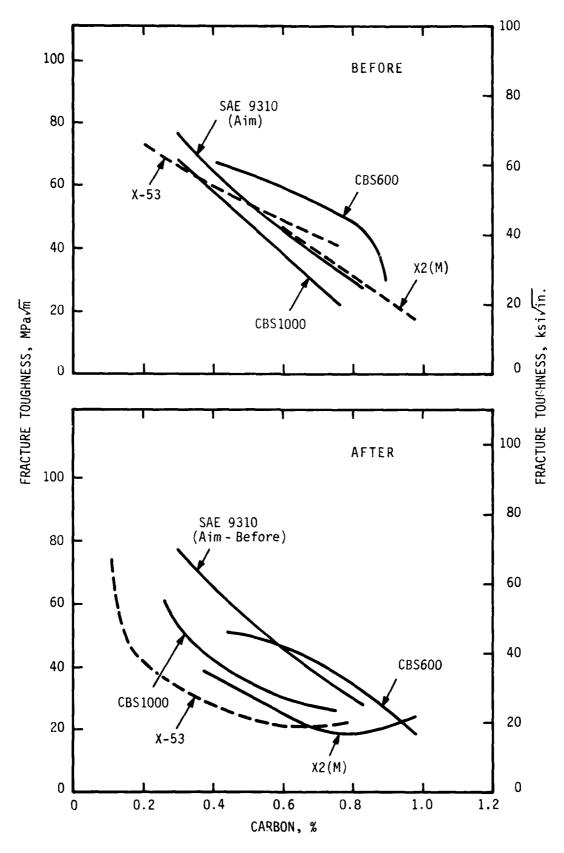


Figure 11 Fracture Toughness at Various Carbon Contents for Candidate Steels Compared to SAE 9310 (Aim). (Residual stress effects included.)

APPENDIX A

Carburizing, Hardening, and Tempering Heat Treatments of Commercial Candidate Steels

CBS600:

Carburized 7 hours at 925 C (1700 F) in an atmosphere carbon potential of 1.10%, cooled to 790 C (1450 F), quenched directly into agitated 65 C (150 F) oil, refrigerated at -80 C (-115 F) for 16 hours and double tempered at 150 C (300 F) for 2 + 2 hours (refrigerated between tempers).

CBS1000

Preoxidized specimens at 950 C (1750 F) for 1 hour, carburized 7 hours at 925 C (1700 F) in an atmosphere carbon potential of 1.10%, quenched directly into agitated 65 C (150 F) oil, reheated to 650 C (1200 F) for 1 hour, air cooled, reheated to 660 C (1220 F) for 1/2 hour, heated to 1010 C (1850 F) for 20 minutes, quenched directly into agitated 65 C (150 F) oil, refrigerated 3 hours at -80 C (-115 F), and double tempered at 315 C (600 F) for 2 + 2 hours (refrigerated between tempers).

X-53:

Preoxidized specimens at 950 C (1750 F) for 1 hour, air cooled, carburized 8 hours at 870 C (1600 F) in an atmosphere carbon potential of 1.10%, quenched directly into agitated 65 C (150 F) oil, reheated to 650 C (1200 F) for 1 hour, air cooled, reheated to 675 C (1250 F) for 1 hour, heated to 1010 C (1850 F) for 1 hour, quenched into agitated 65 C (150 F) oil, refrigerated at -80 C (-115 F) for 3 hours and double tempered at 315 C (600 F) for 2 + 2 hours (refrigerated between tempers).

X2(M):

Preoxidized specimens at 980 C (1800 F) for 1 hour, air cooled, carburized 10.4 hours at 925 C (1700 F) in an atmosphere carbon potential of 1.10%, hardened, carburized 4 hours at 925 C (1700 F) in an atmosphere carbon potential of 0.9%, quenched directly into agitated 65 C (150 F) oil, reheated to 690 C (1280 F) for 1 hour, air cooled, reheated to 675 C (1250 F) for 1/2 hour, heated to 1010 C (1850 F) for 30 minutes, quenched directly into agitated 65 C (150 F) oil, refrigerated at -80 C (-115 F) for 3 hours, and double tempered at 315 C (600 F) for 2+2 hours (refrigerated between tempers).

SAE 9310:

Carburized 7 hours at 925 C (1700 F) in an atmosphere carbon potential of 1.05%, cooled to 840 C (1550 F), quenched directly into agitated 65 C (150 F) oil, refrigerated at -80 C (-115 F) for 16 hours, and double tempered at 150 C (300 F) for 2 + 2 hours (refrigerated between tempers).

APPENDIX B

Carburizing, Hardening, and Tempering Heat Treatments of Experimental Steels

Heats P2622A and P2622B:

Preoxidized at 980 C (1800 F) for 1 hour and carburized 5 hours at 925 C (1700 F) in an atmosphere carbon potential of 1.1% plus 3 hours in a carbon potential of 1.0% and air cooled. Reheated to 1010 C (1850 F) for 1/2 hour and quenched into agitated 65 C (150 F) oil, and double tempered at 315 C (600 F) for 2 + 2 hours.

Heats P2623A and P2623B:

Same as above except steels were refrigerated at -80 C (-115 F) for 16 hours prior to double tempering.

Heats P2624A and P2624B:

Preoxidized at 980 C (1800 C) for 1 hour and carburized 5 hours at 925 C (1700 F) in an atmosphere carbon potential of 1.1% plus 3 hours in a carbon potential of 1.0% and air cooled. Reheated to 1010 C (1850 F) for 1/2 hour and quenched into agitated 65 C (150 F) oil and refrigerated at -80 C (-115 F). Recarburized 4 hours at 925 C (1700 F) in an atmosphere carbon potential of 0.85% and quenched directly into agitated 65 C (150 F) oil, refrigerated at -80 C (-115 F) for 16 hours and double tempered at 315 C (600 F) for 2 + 2 hours (refrigerated between tempers).

